# PATENT SPECIFICATION

NO DRAWINGS

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1,171,971

1,171,971

THE PATENT OFFICE LONDON

Date of filing Complete Specification: 11 Jan., 1968.

Application Date: 16 Jan., 1967. No. 2262/67.

Complete Specification Published: 26 Nov., 1969.



Index at acceptance: —B5 N(17Y, 17X, 20Y, 214, 22Y, 22X, 227, 241, 250, 252Y, 254Y, 255Y, 256, 252X, 265Y, 280Y, 282Y, 283Y, 284Y, 280X, 282X, 290Y, 291Y, 297Y, 298Y, 344, 346, 348, 35Y, 35X, 350, 353, 354, 355, 55Y, 565, 567, 569, 570, 596, 616, 641, 655, 659, 66Y, 66X, 663, 665, 669, 67X, 670, 674, 679, 68X, 69Y, 690, 71X, 710, 712, 713, 715, 750, 79Y, 790, 796)

International Classification: —B 32 b 3/24

## COMPLETE SPECIFICATION

## Improvements in or relating to Non-Woven Structures

We, IMPERIAL CHEMICAL INDUSTRIES LIMITED, Imperial Chemical House, Millbank, London S.W.1, a British Company do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The invention relates to methods for making non-woven fibrillar structures and to the non-woven fibrillar structures made thereby.

Non-woven structures consisting of layers of fibrillated oriented polymeric film adhesively bonded together are known but the methods of fibrillating the layers used hitherto such as flexing, heating and shredding have the disadvantages that either complicated fibrillating equipment is required or that the products produced thereby consist of long strands of film with little coherence between adjacent strands and hence the structures lack dimensional stability. We have found a novel method of fibrillating layers of oriented polymeric film with little coherence between adjacent easily operable equipment and which produces very cohesive fibrillated structures.

Accordingly in one of its aspects, the invention comprises a process for producing a non-woven fibrillar structure comprising superposing a plurality of layers of oriented fibrillatable polymeric film and needle-punching the structure so formed in a needle-loom such that the layers are fibrillated.

In another of its aspects, the invention comprises a non-woven fibrillar needle punched structure comprising a plurality of layers of fibrillated oriented polymeric film bonded together.

The fibrillatable film used for the invention will generally be oriented predominantly either along its length or across its width so that it will fibrillate easily on impact in the direction of predominating orientation. To provide a structure of substantially isotropic properties, the layers of film may be superposed so that their directions of predominating orientation are at such angles as to produce a balanced structure and a convenient and preferred method of arranging this is to superpose the layers of film so that adjacent layers have their directions of predominating orientation at right angles to one another. Alternatively the layers may be arranged to give any desired non-isotropic properties.

Any polymeric film that can be oriented predominantly in one direction and is thus fibrillatable can be employed in this invention, typical films being, for example, those of polyolefins such as polyethylene, polypropylene and poly - 4 - methyl - pentene - 1; polyesters such as poly(ethylene terephthalate); polyamides such as poly(hexamethylene adipamide); poly(vinyl chloride); poly (vinylidene chloride); copolymers of vinyl chloride and vinylidene chloride; and any of these polymers may contain additives to enhance fibrillation. Generally, however, from the point of view of economy and fibrillating properties, polypropylene is the preferred polymer. The superposed film structure may consist of layers of one type of polymeric film only or of several different types.

The type of fibrillated structure produced depends upon the needlepunching conditions to which it is subjected. The needle-punching may be sufficient only to fibrillate the layers

without producing any intermingling of fibrils between layers, in which case further means are necessary to bond the layers together, or a more severe needlepunching treatment can be given which causes fibrils from the various layers to be intermingled and interlocked and, depending on the amount of intermingling may make the use of further bonding means unnecessary. Generally we have found that structures of the former type can be produced by needling lightly, say not more than 600 punches per square inch, with either barbed or unbarbed needles to a moderate depth, whereas structures of the latter type require heavier needling say more than 600 punches per square inch, preferably 1000 punches per square inch with barbed needles and with at least 3 barbs of each needle passing through the structures. The structures may be advanced one or more times through the needle-loom depending upon the properties required and the operating conditions of the needle-loom and the structures may be inverted between passages through the loom so that both sides of the structure are subjected to the needling action and/or they may be passed through a double-bed needle-loom.

If further means are necessary to bond the layers of film together an adhesive may be used and the layers bonded either before or after fibrillation. Preferably however the adhesive whether in its adhesive state or not should be applied to the structure before fibrillation. Any type of adhesive may be used, for instance a latex or a thermoplastic powder, but we have found that adhesives in the form of further layers of film which may be rendered adhesive are particularly useful, a typical film being one of polyethylene. Elastomeric bonding agents may be advantageously used to give useful effects and another convenient method of adhesive bonding is to use films formed from a dispersion of one polymer in another such that at least one of the polymers will form fibrils and the other will act as an adhesive.

Extrusion laminated fibrillatable films in which the components are chosen so that one component can be rendered adhesive without affecting the properties of the other are especially useful in carrying out the present invention since the adhesive component tends to remain associated with the remainder of the fibrils and spread of adhesive into the interstices of the structures, which occurs with other adhesives, is avoided. The use of such films also enables crimped fibrils to be produced if required by suitable choice of components and application of a suitable crimp activating treatment, for instance a heat or solvent treatment. The presence of such crimped fibrils improves the handle of the structures and increases coherency since the crimped fibrils entangle to a greater extent than do uncrimped fibrils.

The only limitation to the particular adhesive used is that it should be such that any treatment, such as a heat or solvent treatment, to render it adhesive should not have any deleterious effect on the fibrils.

Crimped fibrils to enhance cohesion and improve handle can also be produced by using films which have been subjected to, say, a difference in properties across the cross-sections of the film, which difference causes crimp to be developed on application of a suitable treatment.

According to another aspect of the present invention the layers of fibrillatable film can advantageously be associated with webs of fibres or filaments before fibrillation so that some of the fibres extend at least partially through the structure when fibrillated, thus being firmly anchored in the structure and enhancing the resistance to delamination and improving the handle of the structures. The layers of web and film can be arranged in any desired manner but generally speaking it is preferable to have webs on at least one surface of the structures in order to take full advantage of the beneficial effect on handle imparted by the presence of the fibre webs. In this latter case it is also preferable to choose the operating conditions under which the structures are fibrillated so that fibrils do not penetrate the outer surfaces of the webs on the outside of the structure and this can generally be effected by adjusting the depth and amount of needle punching in accordance with the thickness of the structures. We have found that preferably to avoid fibrils appearing on the surfaces of the web not more than five barbs of each needle in the needle looms should pass completely through the structure.

The fibres used to form the webs may be in the form of staple fibres or continuous filaments of any type, for example woven, flax, cotton, silk, regenerated cellulose, mineral fibres, glass fibres and synthetic polymeric fibres (for example polyamide, polyester or polyolefin fibres including fibres derived from split film). The webs may be prepared by a variety of methods, the method selected depending to a great extent on the length of the fibres used. Staple fibre and continuous filament webs can be prepared by air laying processes, for example, and staple fibre webs may be prepared by carding and cross-laying techniques. The webs may include a proportion of fibres which can be rendered adhesive to enhance resistance to delamination and anchoring of the webs and such fibres may be homogeneous binder fibres or composite fibres containing a component which can be rendered adhesive without affecting the remainder of the fibres.

It is well known to produce non-woven fibrous structures by forming webs of natural or synthetic fibres and needle-punching the webs in needle-looms. In addition, it has often

been found advantageous to reinforce such structures with, for example, woven structures incorporated within the webs. The processes for producing such structures are, however, often complicated, time-consuming and expensive since satisfactory webs have to be carefully prepared and satisfactory reinforcing fabrics have to be produced. Our discovery that fibrillatable films can be successfully and easily fibrillated in a needle-loom provides a process for producing non-woven fibrous structures which overcomes the aforesaid disadvantages and provides products having superior properties. Thus structures made according to the present invention have advantages over prior art structures consisting only of fibrous webs or fibrous webs reinforced with non-fibrillated film in that an elastic stretch is imparted to the structures, for instance, in the case of layers of oriented film superposed with their directions of orientation at right angles, along the diagonal lines bisecting those directions of orientation, which makes them more similar to woven or knitted fabrics. The products of the invention are suitable as, amongst other things, filtration media, sacking and tarpaulin materials, carpet backings and floor coverings and may be moulded to any desired shape.

The invention will now be described in more detail with reference to the following examples which are in no way intended to limit the scope of the invention.

EXAMPLE 1

Sample of three composite structures, one consisting of eight layers of uniaxially drawn polypropylene film 0.07 mm thick superimposed with the directions of orientation of adjacent layers at right angles, a second consisting of six layers and the third consisting of four layers similarly arranged were forwarded through a needle-loom under a series of different conditions. The needle-loom was equipped with 32 gauge needles having 0.25" of barb free length from their points and 9 barbs spaced equally part along approximately 0.675 inches of their length. A first set of the three samples was punched to a needle depth of 0.5", i.e. 0.5" of needle passed below the bottom layer of film, and a punch density of 300 p.p.s.i. and the samples were then inverted and the process repeated. The layers of film were clearly fibrillated by the action of the needles but in all three samples coherence between the layers was poor. A second set of the samples was then punched to a needle depth of 0.5" and a punch density of 1000 p.p.s.i., inverted and the process repeated. In this case there was good intermingling between fibrils from the various layers and the finished fibrillated structures were very coherent in all samples and useful as a filter materials. A third set of samples were punched to a needle depth of 0.25" and a punch density of 1000 p.p.s.i., inverted and the process repeated and in all three cases the layers were heavily fibrilated but there was little coherence between layers. A fourth set of samples were then punched to a depth of 0.375" and a punch density of 1000 p.p.s.i., inverted and the process repeated and once again although the layers were heavily fibrillated there was little coherence between layers with all three samples.

Thus, in order to produce satisfactory coherent structures without application of any adhesive bonding from the samples used, a needle depth of about 0.5" and a punch density of more than 500 p.p.s.i. are necessary.

EXAMPLE 2

Two layers of oriented polypropylene film 0.07 mm thick were superposed so that their directions of predominating orientation were substantially at right angles, with a layer of polyethylene powder sandwiched between them. The structure was heated in an air oven at a temperature of 130°C to fuse the polyethylene powder and bond the film together and the structure was then needle-punched in a needle-loom to a density of about 600 punches per square inch. The resultant fibrillated products are suitable as sacking or tarpaulin material.

EXAMPLE 3

Two layers of uniaxially oriented fibrillatable polypropylene film 0.07 mm thick were superposed so that their directions of predominating orientation were substantially at right angles with a layer of 0.002" thick polyethylene film separating them. The structure was needle punched to 600 p.p.s.i. (300 p.p.s.i. each side) with 32 gauge barbed needles at 0.025" depth. The structure was hot pressed at 400 p.s.i. and 135°C and a coherent fibrillated product suitable as a packaging material resulted.

EXAMPLE 4

Example 3 was repeated with a polyester-polyurethane elastomer film replacing the polyethylene film and the heat pressing temperature at 145°C. The resultant fibrillated product possessed useful elastomeric properties and was again suitable, for instance, as a packaging material.

EXAMPLE 5

Six layers of oriented polypropylene film 0.07 mm thick were superposed such that the direction of predominating orientation of each layer was substantially at right angles to that of its adjacent layers and the structure was needle-punched in a needle-loom to a density of about 1000 punches per square inch. The resultant product was a coherent assembly suitable as a filtration medium.

EXAMPLE 6

Four layers of an extrusion laminated film, 0.07 mm thick and consisting of a layer of

polyethylene and a layer of polypropylene and uniaxially oriented, were superimposed and needlepunched with 32 gauge needles as described in example 1 to a depth of 0.5" and a punch density of 1000 p.p.s.i., inverted and the process repeated. The structure was then heated to a temperature of 135°C in a hot air oven, which treatment caused fibrils in the structure to crimp and the polyethylene to become adhesive and bond the fibrils and the layers more firmly together.

EXAMPLE 7

Samples consisting of four layers of uniaxially oriented polypropylene film 0.07 mm thick superposed with the directions or orientation of adjacent layers at right angles, were sandwiched between layers of 4 oz/sq. yd. non-woven web made from 3 denier poly-(hexamethylene adipamide) fibres and one of the samples was punched in a needle-loom fitted with 32 gauge needle to a punch depth of 0.5" and a punch density of 1000 p.p.s.i., inverted and the punching process repeated. Inspection showed that fibrils from the various film layers were intermingled, the product was a very coherent structure suitable as a carpet backing, and no fibrils from the film layers had been carried through to the outer surfaces of the webs.

A second sample was similarly needle-punched but this time to a needle depth of 0.75". Some fibrils from the film layers this time appeared on the outer surfaces of the web detracting somewhat from the handle of the structure.

A third sample was similarly needle punched, this time to a needle punch depth of 0.25". No fibrils appeared on the outer surfaces of the web and the structure was quite coherent, the fibrillated layers being held together by the fibres passing through the layers.

EXAMPLE 8

Two layers of oriented polypropylene film 0.07 mm thick were superposed such that their directions of predominating orientation were substantially at right angles and a sheet of unoriented polyethylene film was placed between them. The assembly was laid on a 2 ounces per square yard non-woven web of polypropylene fibres produced on a PROCTOR and SCHWARTZ "Duo-form" air laying machine and a similar web was laid on top of the structure. The assembly was needle-punched in a needle-loom to a density of 300 punches per square inch and then reversed and punched again to the same density. The needled structure was lightly calendered at a temperature of 135°C to fuse the polyethylene film and the resultant product was suitable as a primary carpet backing.

EXAMPLE 9

A polypropylene and polyethylene film laminate 0.6 mm thick was produced from a double extrusion die and oriented predominately in one direction. The laminate was superposed on a layer of oriented polypropylene film such that the polyethylene was sandwiched between the two polypropylene layers and the directions of predominating orientation of the polypropylene layers were substantially at right angles to one another. The assembly was laid on a 1.5 ounces per square yard non-woven web of 3 denier 2-1/2 polyhexamethylene adipamide fibres produced on a PROCTOR and SCHWARTZ "Duo-form" air laying machine and a similar web was laid on the top of the structure. The assembly was needle-punched to a density of 300 punches per square inch in a needle-loom and then reversed and punched again to the same density. The needled structure was lightly calendered at a temperature of 140°C to fuse the polyethylene film leaving a product which was suitable as a primary carpet backing.

EXAMPLE 10

Two layers of oriented polypropylene film 0.07 mm thick was superposed such that their directions of predominating orientation was substantially at right angles and the layers were laid on a 5 ounces per square yard non-woven web of composite fibres having two components in equal proportions arranged side-by-side, one of the components being poly-(hexamethylene adipamide) and the other being a 70/30 random copolymer of hexamethylene adipamide and epsilon caprolactam, the web being produced on a PROCTOR and SCHWARTZ "Duo-form" air laying machine. A similar web was laid on the top of the structure and the assembly was needle-punched in a needle-loom to a density of 300 punches per square inch. The assembly was reversed and punched again to the same density and heated in a pressure steam chamber at 35 psig. to fuse the copolymer component of the composite filaments and also cause the filaments to crimp. The resultant product was suitable as a hard wearing floor covering.

EXAMPLE 11

Two layers of uniaxially oriented polypropylene film 0.7 mm thick with their directions of orientation at right angles were sandwiched between 2 oz/sq. yard non-woven webs of 3 denier polypropylene staple fibres, the webs each having 2 oz/ sq. yd. layers of polyethylene powder sintered to the sides nearest the film. The structure was needle-punched with 32 gauge needles as used in example 1, to a depth of 0.5" and a punch density of 1000 psi, inverted and the needle punching action repeated. The resulting product which had no fibrils on its surface was calendered between hot rolls at 145°C to render adhesive the polyethylene powder and

produce a very coherent material suitable as a carpet backing.

EXAMPLE 12

Two layers of uniaxially oriented extrusion laminated film 0.7 mm thick and consisting of a layer of polypropylene and a layer of polyethylene were sandwiched between 2oz/sq. yd. webs of 3 denier polypropylene fibres and needlepunched as in example 11 so that no fibrils appeared on the surfaces of the web. The structure was then heated in an air oven at 135°C to cause the fibrils to crimp and the polyethylene component to become adhesive. The resulting product was a very drapable, coherent, well bonded structure.

WHAT WE CLAIM IS:—

1. A process for producing a non-woven fibrillar structure comprising superposing a plurality of layers of oriented fibrillatable polymeric film and needlepunching the structure so formed in a needle-loom such that the layers are fibrillated.

2. A process as claimed in claim 1 in which the needlepunching action causes fibrils in different layers to become entangled and interlocked.

3. A process as claimed in claim 2 in which the structures are needle punched with barbed needles to a punch density greater than 600 p.p.s.i. and at least 3 barbs from each needle pass completely through the structure.

4. A process as claimed in claim 1 in which the needlepunching action is insufficient to cause fibrils in the different layers to become substantially entangled and interlocked.

5. A process as claimed in any of claims 1 to 4 in which one or more webs of fibres or filaments are assembled with the layers of film and the composite structure needle-punched.

6. A process as claimed in claim 5 in which the layers of film are sandwiched between layers of fibre-webs.

7. A process as claimed in claim 6 in which the needlepunching treatment does not produce fibrils on the outer surface of the webs.

8. A process as claimed in claim 7 in which not more than five needle-barbs from each needle pass completely through the structure.

9. A process as claimed in any one of the preceding claims in which films in adjacent layers are superposed with their directions of predominating orientation at right angles.

10. A process as claimed in any of the preceding claims in which at least one of the fibrillatable film layers possesses potential crimp and the structure is subjected, after needle-punching, to a treatment to develop crimp in the fibrils.

11. A process as claimed in any of the preceding claims in which an adhesive for bonding together the film layers is incorporated in the structure.

12. A process as claimed in claim 11 in which the adhesive is incorporated in the structure in a potential adhesive state and the structure is subjected to a treatment to develop the adhesive characteristics.

13. A process as claimed in claim 12 in which said adhesive is incorporated in the structure before fibrillation and said treatment is carried out after fibrillation.

14. A process as claimed in either of claims 12 or 13 in which said adhesive is incorporated as a thermoplastic powder.

15. A process as claimed in any of claims 12 to 13 in which said adhesive is incorporated as a thermoplastic film.

16. A process as claimed in claim 15 in which said film is incorporated as an extrusion laminated fibrillatable film having at least one fibrillatable component thermoplastic and a component which can be rendered adhesive without affecting the properties of the remainder of the film.

17. A process as claimed in claim 16 in which the film content of the structure is totally assembled from said extrusion laminated fibrillatable films.

18. A process as claimed in any of claims 14 to 17 in which the thermoplastic material is polyethylene.

19. A process as claimed in any of claims 11 to 13 in which said adhesive is incorporated as thermoplastic potentially adhesive fibres.

20. A process as claimed in claim 19 in which the potentially adhesive fibres are incorporated as composite fibres containing a component which can be rendered adhesive without affecting the properties of the remainder of the fibres.

21. A process as claimed in any of the preceding claims in which the structure is assembled from polypropylene film.

22. Process substantially as described herein and with particular reference to the foregoing examples.

23. A non-woven needle-punched fibrillar structure comprising a plurality of layers of fibrillated oriented polymeric film bonded together.

24. A non-woven fibrillar structure as claimed in claim 23 in which fibrils from different layers are entangled and interlocked thus bonding the structure together.

25. A non-woven fibrillar structure as claimed in claim 23 in which there is no entanglement between fibrils of different layers.

26. A non-woven fibrillar structure as claimed in any of claims 23 to 21 which contains one or more webs of fibres needlepunched to the layers of film.

27. A non-woven fibrillar structure as claimed in claim 26 in which the layers of

film are sandwiched between layers of fibre-webs.

28. A non-woven fibrillar structure as claimed in claim 27 in which no fibrils appear on the surfaces of the webs.

29. A non-woven fibrillar structure as claimed in any one of claims 23 to 28 in which the predominating directions of orientation in adjacent layers of film are at right angles.

30. A non-woven fibrillar structure as claimed in any of claims 23 to 29 in which at least some of the fibrils are crimped.

31. A non-woven fibrillar structure as claimed in any one of claims 23 to 30 in which the structures are adhesively bonded.

32. A non-woven fibrillar structure as claimed in claim 31 in which the adhesive is a thermoplastic powder.

33. A non-woven fibrillar structure as claimed in claim 31 in which the adhesive is a thermoplastic film.

34. A non-woven fibrillar structure as claimed in claim 33 in which said film is an extrusion laminated film having at least one fibrillatable component and a thermoplastic component capable of being rendered adhesive without affecting the properties of the remainder of the film.

35. A non-woven fibrillar structure as claimed in claim 34 in which the total film content of said structure comprises said extrusion laminated film.

36. A non-woven fabrillar structure as claimed in any one of claims 32 to 25 in which the thermoplastic material is polyethylene.

37. A non-woven fibrillar structure as claimed in claim 31 in which said adhesive is in the form of thermoplastic fibres.

38. A non-woven fibrillar structure as claimed in claim 37 in which said fibres are composite fibres containing a component capable of being rendered adhesive without affecting the properties of the remainder of the fibres.

39. A non-woven fibrillar structure as claimed in any one of claims 23 to 38 in which said fibrillatable film is polypropylene.

40. Non-woven fibrillar structures substantially as described herein and with particular reference to any of the foregoing examples.

41. Non-woven fibrillar structures when made by the processes of any of claims 1 to 22.

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Printed for Her Majesty's Stationery Office by the Courier Press, Leamington Spa, 1969.
Published by the Patent Office, 25, Southampton Buildings, London, W.C.2, from which copies may be obtained.